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CATALYST RESEARCH AND DEVELOPMENT FOR INDUSTRIAL APPLICATIONS

DR/DRK/81/013/A/01/37 DEMOCRATIC PEOPLE'S REPUBLIC OF KOREA

Final Report

Based on the work of Radko Komers, expert in R&D Equipment for Catalytic Chemistry and Carlos E. Gigola, expert in Catalytic Hydrogenation

United Nations Industrial Development Organization Vienna

INTRODUCTION

Arrival at Pyongyang airport, Democratic People's Republic of Korea, took place in the morning of February 13, 1984. We were welcomed there by Mr. Pak Yong Son, from the Institute of Analytical Chemistry of Hamhung, by Mr. Jo Myong Grun, officer of the Department of Foreign Affairs of the Academy of Science and by Mr. Pek Rak Bon, of the Institute of Organic Chemistry (IOC) in Hamhung. The former man was able to communicate with us in English and from that moment his cooperation was absolutely essential to accomplish our mission.

On the same day we visited the UNDP office where we met Mr. Slobodan Ristic, the Resident Representative, and Mr. Richard Millar his Deputy. It was decided that we should depart immediately to our duty station, Hamhung and complete our missio. on February 21. We arrived there the next day and were introduced to the Deputy Director of IOC and his staff (see Apendix I). We learnt about the activities, organization and main lines of applied research at IOC.

In the afternoon a more detailed explanation was given on research projects that have high priority for the development of the national chemical industry which cannot be carried out without external support. The three laboratories where research on catalysis have been presently done were visited the same day.

On February 15, during the morning a lecture was given by Carlos E. Gigola on "Selective Hydrogenation of Acetylene in the presence of Ethylene" and was followed by Radko Komers' lecture on "Study Catalysis by Temperature-Programmed Desorption". Approximately 25 members of IOC were present and participated in the subsequent discussion. In the afternoon after visiting the library we met the heads of each catalytic laboratory in crder to evaluate the present state of their projects, what results have been obtained so far, problems encountered and experimental difficulties, etc.

On February 16 a meeting took place to discuss technical problems between the mission members and the IOC staff. The next day we were introduced to the chief engineers of a Petroleum Refinary and a Petrochemical Company (see Apendix I). The members of the mission were also invited to visit several laboratories of the Institute of Analytical Chemistry on February 18. On Sunday, February 19, a visit to the Vinalon complex was arranged.

In addition to the above mentioned activities the collected information was used to write the present report. The main conclusions and recommendations are given below.

I. GENERAL EVALUATION OF IOC CATALYTIC LABORATORIES

A. <u>Facilities</u>

The IOC includes three laboratories dealing with catalytic research and one laboratory for analytical service. To our opinion the present physical plant is suitable to accommodate the present staff and equipment. It will be also sufficient for new experimental work.

B. Personnel

Eighty persons are available for the laboratories mentioned above. Out of this number, 10 are PhD and MsS, 30 are graduates from universities or colleges and 40 persons are technicians. The level of knowledge of the educated staff seems to be reasonable but there is still some need for training in special areas concerning catalytic research, e.g., reaction kinetics, thermodynamics, catalysts preparation, characterization of solid surfaces, etc.

C. Equipment

The equipment available so far is almost obsolete and it is not adequate to carry out the pretentious research projects. Only simple glass apparatuses, laboratory reactors, standard labware and homemade gas chromatographs were seen during our visit, but an all metal medium pressure reactor (40-50 atm) is in operation.

D. Research

The level of research was evaluated to limited extend because reports or papers were not available in English. However, we realized from discussions that many valuable results have been obtained up to now. For example the development of new Ag/SiO_2 catalyst for the production of ethylene oxide was partly performed in IOC. This reflects the capability and skills of the present staff. Therefore we can anticipate that similar problems will be successfully solved too.

E. Library

The large collection of books is printed mainly in Japanese language and just a few of them are modern. English literature is limited and material concerning the most recent advances in catalytic research is missing. With regard to periodicals we can say that the situation seems to be better. Most common and important journals were found in the library.

F. Relation between IOC and the industry

A meeting was arranged among representatives of IOC, the members of the UNIDO mission and chief engineers of Sangri Petroleum Refinary and Namhung Petrochemical Company.

The Sangri Refinary is located in La-Zin approximately 600 Km north of Hamhung, near the USSR border. It produces gasoline, diesel oil, benzene, toluene, LPG, lubricants, etc. by various catalytic processes. Most catalysts are imported from the USSR but the Korean government decided to reverse this situation in the near future. Benzene production is expected to be increased by the catalytic dealkylation of xylenes and toluene. This important process is to be developed in close cooperation with catalytic groups of IOC.

The Namhung Petrochemical Company produces a variety of chemicals from naphta pyrolisis. The main products are: polyacrylonitrile, polyethylene, ethylene oxide and others. The catalyst for the production of acrylonitrile consists of Sn-Sb oxides and is manufactured on the spot. The enhancement of the acrylonitrile production is of significant importance to the country economy, so a better catalyst is needed. This project is being investigated by an IOC catalytic laboratory.

In both cases the applied research done in IOC will have a desirable impact on the production.

II. SELECTED AREAS OF RESEARCH

The catalytic processes play an important role in modern chemical industry. Therefore it is advisable to have national research facilities strong enough to provide technological assistance for existing processes and to develop new ones.

According to the information given in the previous section of this report the catalytic laboratories of IOC could be considered to be in such a position provided that the present capabilities will be strengthened. Taking into account the high demand for basic chemicals, the government policy not to be dependent on imports and the present technological problems in the local industry, the following three main research projects have been selected to be carried out in IOC.

A. Project 1 - Methanol Conversion to Olefines

<u>Head</u>: Mr. Ryom Gyang Jae
 <u>Personnel</u>: 1 PhD; 2 MsS; 8 Grd; 7 Tech.

- 2) Objective of the research: The DPRK has no oil or natural gas that can be used for the production of olefines. This situation is to be solved by using CH_3OH as an alternative starting material. Actual production of CH_3OH is 50,000 ton/year and it will be increased by 40-50% in 1985. The purpose of this project is to develop a proper zeolite catalyst for this reaction.
- 3) <u>Present state</u>: Several zeolite catalysts of the ZSM type has already been prepared and tested. Using a glass reactor at atmospheric pressure they have obtained reasonable conversion to ethylene but the selectivity must be significant increased. These results have been already published in the Korean Bulletin of Chemistry and another paper is ready for publication.
- 4) <u>Planned activities</u>: In order to test catalysts under industrial conditions it will be necessary to have a high pressure reactor (100 atm). Modern characterization techniques should be introduced in order to improve the selectivity and life time. New catalysts will be prepared and tested.

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5) Future_needs:

a) Equipment and supplies: see Appendix II.

- b) Study Tours: a 4 week study tour is proposed for Mr. Choi Yan Thik.
- c) Training of personnel: 16 month/man of training are required for 2 men, starting as soon as possible.
- 6) Special recommendations: With regard to training it is advisable to select a laboratory or institution where the research involves the use of zeolites and high pressure experimentation. The USSR Academy of Sciences and the Czechoslovak Academy of Science should be consulted. A good laboratory for zeolite research is at the Ecole Superieur de Chimie de Montepellier, France, under the supervision of Prof. Francois Figueras. Also the Institute of Catalysis in Lyon, France, is recommended, both for study tours and training. To our opinion, Mr. Ji Dong Han is a good candidate for training.

B. Project 2 - Catalytic Oxidation

<u>Head</u>: Dr. Kim Hyong Gi
 <u>Personnel</u>: 1 PhD; 3 MsS; 7 Grd; 11 Tech.

- 2) Objective of research: The production of acrylonitrile for synthetic fibers is based on the ammoxidation reaction of propilene. A plant located in Namhung uses an Sb-Sn oxide catalyst which was originally imported from France and now is manufactured in DPRK. In order to increase the production of acrylonitrile it is necessary to improve the catalyst activity and selectivity.
- 3) <u>Present State</u>: Using a flow reactor they have obtained a selectivity of 60-65% which should be increased to 71-75% to achieve the project objective. Experimental difficulties are encountered with the present reactor. The results obtained up to now have already been published in a local journal.
- 4) <u>Planned activities</u>: Attention should be focused on the optimization of the present catalyst by the addition of promotors as Fe or Cu. Another possibility is to develop a new Mo-Bi/ SiO₂ catalyst. In order to improve the experimental set up it will be advisable to have a recirculation reactor with a

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certain degree of automation.

- 5) Future needs:
 - a) Equipment, supplies and books: see Appendix III.

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- b) Study Tours: a 4 week study tour is proposed for the project head, Dr. Kim Hyong Gi.
- c) Training of personnel: 32 months/man are required for 4 persons starting as soon as possible.
- 6) <u>Special recommendations</u>: In order to find convenient places for training and study tours we suggest to follow the recommendations of Mrs. Raissa Tchesnokova and Mr. Aiaz Efendiev, members of this mission.

C. Project 3 - Catalytic Dealkylation

- 1) <u>Head</u>: Dr. Kim Jong Taek
 - Personnel: 2 MsS; 11 GRD; 7 Tech.
- 2) Objective of the research: The primary motivation for doing this project is the high demand of benzene which could be obtained by the dealkylation of toluene and xylenes. These two chemicals are supplied by a petroleum refinary located in the northern part of DPRK. This technology is based on the utilization of a supported Cr_2O_3 catalyst.
- 3) <u>Present State</u>: A high pressure all metal apparatus which is unsatisfactory has been set up and used for testing different samples of a $\operatorname{Cr}_2O_3/\operatorname{Al}_2O_3$ catalyst. While the activity and selectivity of the catalysts are satisfactory the life time should be significantly extended. Four papers have already been published in the Korean Journal of Chemistry and Chemical Technology.
- 4) <u>Planned activities</u>: Modern characterization techniques and a high pressure all metal apparatus should be introduced for the evaluation of new catalysts samples in order to obtain reproducible results and to facilitate its interpretation.
- 5) Future needs:
 - a) Equipment, supplies and books: See Appendix IV.
 - b) Study tours: a 4 week study tour is proposed for Mr.Choi Yan Thik.
 - c) Training of personnel: 24 months/men of training are

required for 3 men starting as soon as possible.

6) <u>Special recommendations</u>: The Deputy Director of IOC mentioned to us that they would prefer to send people for training or study tours to the USSR, German Democratic Republic or other socialist countries. Consequently it is advisable to contact the Academies of Sciences in those countries. However we could also suggest the Institute of Catalysis in Lyon, France, and the Université Catholique de Louvain, Louvain la Neuve, Belgium, where Prof. B. Delmon should be contacted.

III. SPECIAL RECOMMENDATIONS

During our mission we encountered serious difficulties to communicate with the IOC staff due to the fact that English is not spoken. Thank Mr. Pak Yong Son from the Institute of Analytical Chemistry who provided full translation service, we were able to gather all information necessary to make the mission possible. Similar service was produced by Mr. Pek Rak Bon from IOC for Russian interpretation. For these reasons we strongly recommend to arrange courses in English for the research personnel.

In addition to the short term training necessary for specific projects we advise to send young promising graduates abroad to obtain a high level degree (e.g., PhD, CSc).

To our knowledge there is no any electronic data processing system available for calculating, data acquisition and/ or process control purposes. We suggest to incorporate such an equipment into IOC depending on the future budget. The same criteria should be followed with purchasing of modern tools for the characterization of solids, e.g., Autopor 9200 (Micromeritics, U.S.A.). Such an apparatus automatically provides the surface area, He-density, Hg-density, pore size distribution and other measurements.

Finally, taken into account the need for analytical equipment and services to carry out the projects, a close interaction between the catalytic laboratories and the Institute of Analytical Chemistry is advisable.

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IV. ACKNOWLEDGMENT

We would like to thank Deputy Director of IOC, Mr. Kim Jdung Bai and his staff for their effort to satisfy and coordinate valuable discussions essential to achieve the objectives of this mission. We also acknowledge the continuous performance of both interpreters. Hospitality and kindness of all Korean people we met is also appreciated. Besides we should like to mention good cooperation among all members of the mission. Finally we thank members of UNIDO Chemical Industries Branch in Vienna and members of UNDP office in Pyongyang for their help.

APPENDIX I

Professionals that participated in meetings and discussions - Mr. Kim Jdung Bai; Deputy Director of IOC - Mr. Jo Myong Grun; Officer of Department of Foreign Affairs, Academy of Sciences of DPRK - Mr. Kim Dal Yen; Instructor, Hamhung Branch of the Academy of Sciences - Mr. Pak Yong Son; Institute of Analytical Chemistry, Hamhung - Mr. Ryom Gyang Jae; Head of Catalyst Laboratory, IOC - Mr. Kim Hyong Gi; Head of Catalyst Laboratory, IOC - Mr. Kim Jong Taek; Head of Catalyst Laboratory, IOC - Mr. Ji Dong Hen, IOC - Mr. Ko Yong Y1, IOC - Mr. Choi Yan Thik, IOC - Mr. Pek Rak Bon, IOC - Mr. Sin Sen Gi; Chief engineer, "Sangri" Petroleum Refinary - Mr. O Won Sek; Chief engineer, "Namhung" Petrochemical Company

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APPENDIX II

EQUIPMENT REQUIRED IN PROJECT 1

| Item | Description | Quantity | Estimated cost US\$ |
|------|--|----------|------------------------|
| 1) | X-ray diffractometer with variable temperature sample chamber (up to 1300°C) | 1 | 120,000 |
| 2) | High pressure catalyst ac- tivity measuring device (up to 100 atm) | 1 | 15,000 |
| 3) | Gas chromatograph with FID and accesories | 2 | 30,000 |
| 4) | Vacuum furnace (up to 1200°C) | 2 | 6,000 |
| 5) | Micro calorimeter | 1 | 3,000 |
| | | Total | 174,000 |
| | Books (see page 14) | | 1,000 |

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APPENDIX III

EQUIPMENT REQUIRED IN PROJECT 2

| Item | Description | Quantity | Estimated Cost US\$ |
|------|--|----------|------------------------|
| 1 | Circulation reactor (catalyst activity measurements) | 2 | 10,000 |
| 2 | Pulse flow reactor (catalyst activity measurements) | 2 | 10,000 |
| 3 | Gas chromatograph with FID and accesories | 2 | 30, 000 |
| 4 | Differential thermo analyzer | 1 | 25,000 |
| 5 | Liquid metering pump (1-2 ml/min. | .) 6 | 6,000 |
| 6 | Liquid micro metering pump | 6 | 1,200 |
| 7 | Mercury Porosimeter (up to 4000 at | m) 1 | 5,000 |
| 8 | BET Surface area apparatus | 1 | 2,000 |
| 9 | Accesories for High Vacuum Ad- sorption Apparatus | 1 | 10,000 |
| 10 | Apparatus for measuring mechanica | al | |
| | properties of catalysts | 1 | 2,000 |
| 11 | Cathetometer | 2 | 2,000 |
| 12 | Infrared spectrophotometer | 1 | 20,000 |
| 13 | Analytical balance | 2 | 4,000 |
| 14 | Surface potentiometer | 1 | 1,000 |
| | Το | tal | 128,200 |
| | Books (See page 14) | | 2,000 |

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APPENDIX IV

EQUIPMENT REQUIRED IN PROJECT 3

| Item | | Quantity | Estimated cost US\$ |
|------|---|----------|---------------------|
| 1 | High pressure catalyst activity measuring device (up to 200 atm) | 1 | 10,000 |
| 2 | High pressure hydrogen compres- sor (400 atm; 5 Nm3/h) | 1 | 15,000 |
| 3 | High pressure metering pump (200 atm; 10 lt/h) | 4 | 4,000 |
| 4 | High pressure gas flowmeter (200 atm; 5 Nm3/h) | 4 | 4,000 |
| 5 | High pressure liquid flowmeter (200 atm; 10 lt./h) | 4 | 4,000 |
| 6 | High pressure gas metering pump (200 atm; 0-5 Nm3/h) | 4 | 4,000 |
| 7 | Gas chromatograph with FID and accesories | 3 | 45,000 |
| 8 | Pulse flow reactor (high pressure) | 1 | 5,000 |
| 9 | Programmable furnace (up to 1500°C |) 2 | 8,000 |
| 10 | Dew point meter | 1 | 2,000 |
| 11 | Small size refrigeration unit | 2 | 10,000 |
| 12 | PH Meter | 2 | 2,000 |
| 13 | Analytical balance (micro and semi micro) | 2. | 4,000 |
| 14 | Titration apparatus for precipi- tation reactions (automatic) | 1 | 1,000 |
| 15 | High capacity hydrogen generator 8 Nm3/hr | 1 - | 5,000 |
| | Total | | 123,000 |
| | Books (See page 14) | | 2,000 |

BOOKS AND JOURNALS

BOOKS

- "Spectroscopy in heterogeneous catalysis", Delgass, W.N., G.L. Haller and R. Kellerman - Academic Press.
- "Progress in catalyst deactivation", Figueiredo, J.L. (Ed.) -Martinus Nijhoff.
- "Catalytic conversion of hydrocarbons", Germain, J.E. Academic Press.
- "The chemical physics of solid curfaces and heterogeneous catalysis".Vol. 4 - Ponec, V. - Elsevier Scientific Publ. Co.
- "The chemistry of catalytic hydrocarbon conversions", Pines, H. Academic Press.
- Chemical and physical aspects of catalytic oxidation", Portefaix, J.L.and Figueras, F. - C.N.R.S.
- "Preparation of catalysts.Scientific bases for the preparation of heterogeneous catalysts". 1976, Delmon, B.(Ed.), P.A.Jacobs (Ed.) and Poncelet, G. (Ed.). Elsevier Scientific Publishing Co.
- "Preparation of catalysts II. Scientific bases for the preparation of heterogeneous catalysts". 1979, Delmon, B.; P. Grange;
 P. Jacobs and G. Poncelet. Elsevier Scientific Publishing, Co.
- "Preparation of catalysts III. Scientific bases for the preparation of heterogeneous catalysts". 1983. Poncelet, G.(Ed.); P. Grange (Ed.) and P.A. Jacobs (Ed.). - Elsevier Scientific Publishing Co.
- "Design of industrial catalysts". Trimm, D.L. Elsevier Scientific Publishing Co.
- "The chemical physics of solid surfaces and heterogeneous catalysis. Vol. 1: Clean solid surfaces". King, D.A. and D.P. Woodruf. - Elsevier Scientific Publishing Co.

- "Catalytic hydrogenation in organic synthesis". Rylander, P. Academic Press.
- "Hydrogenation catalysis". Peterson, R. Noyes Data Corporation.
- "Catalysis by zeolites". Imelik, B. (Ed); C. Naccache (Ed.) and Y. Ben Taarit (Ed.). - Elsevier Scientific Publishing Co.
- "Chemistry of catalytic processes". Gates, B.C., J.R. Katzer and G.C.A. Schuit. - McGraw-Hill Book Co.
- "Experimental methods in catalytic research".Vol. 1. Anderson, R.B. (Ed.). - Academic Press.
- 'Experimental methods in catalytic research''. Vol. 2. Anderson, R.B. (Ed.) and P.T. Dawson (Ed.). - Academic Press.
- "Experimental methods in catalytic research". Vol. 3. Anderson, R.B. (Ed.) and P.T. Dawson (Ed.). - Academic Press.
- "Catalysis, Science and Technology". Vol. 1. Anderson, J.R. and M. Boudart. Springer-Verlag.
- "Catalysis, Science and Technology". Vol. 2. Anderson, J.R. and M. Boudart. - Springer-Verlag.
- "Selective oxidation of hydrocarbons". Hucknall, D.J. Academic Press.
- "Structure of metallic catalysts". Anderson, J.R. Academic Press.
- "Catalysis in C₂ chemistry". Keim, W. D. Reidel Publishing Company.
- "Chemicals from synthesis gas". Sheldon, R.A. D. Reidel Publishing Company.

1.

- "Surface properties and catalysis by non-metals". Bonnelle, J.;
 B. Delmon, and E. Derovane. D. Reidel Publishing Company.
- "Catalysis Specialist periodical reports" Vol. 1, 2, 3 and 4. The Chemical Society - Burlington House - London.
- "Chemistry of the metal-gas interface". Roberts, M. and C. McKee Clarendon Press - Oxford - England.

JOURNALS

- "Applied Catalysis" - Vol. 1, 1981 - Vol. 2-3 and 4, 1982. Vol. 5-6-7 and 8, 1983.

Elsevier Science Publishers, Amsterdam, The Netherlands.

